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IS 12107-8 (1987): Methods of chemical analysis of alumino-silicate refractory materials, Part 8: Determination of calcium and magnesium [MTD 13: Ores and Raw Materials]

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UDC 666·762·1 : 543 [546·41 + 546·46] (First Reprint MAY 1989) **IS : 12107 (Part 8) - 1987**

(Reaffirmed 2001)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ALUMINO SILICATE REFRACTORY MATERIALS

PART 8 DETERMINATION OF CALCIUM AND MAGNESIUM

Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee, SMDC 35 [Ref : Doc : SMDC 35 (3008)]

1. Scope — This standard (Part 8) covers a method for determination of calcium and magnesium in alumino silicate refractory materials.

2. Determination of Calcium and Magnesium by EDTA Method

2.1 *Outline of the Method* — An aliquot of man solution R_2O_3 group elements are masked with triethanolamine, calcium-magnesium are determined at *p*H 10 using mixed indicator. In another aliquot, magnesium is precipitated at *p*H 12 in the presence of triethanolamine and calcium is titrated by EDTA using (P&R) indicator. The value of calcium is subtracted from magnesium-calcium, and magnesium is obtained.

2.2 *Reagents*

2.2.1 *Sodium hydroxide* (2 *N*) — Dissolve 40 g of sodium hydroxide in water and dilute to 500 ml.

2.2.2 *Dilute hydrochloric acid* — 30 percent (*v*/*v*).

2.2.3 *Triethanolamine* — 30 percent (*v*/*v*).

2.2.4 *Potassium cyanide solution* — 5 percent (*v*/*v*).

2.2.5 *Hydroxlamine hydrochloride* — 10 percent (*m*/*v*).

2.2.6 *Hexamine solution* — 10 percent (*v*/*v*).

2.2.7 *Buffer solution* (*pH* 10) — Dissolve 67·5 g of ammonium chloride in 250 ml of water; add 570 ml of ammonia solution and dilute to 1 litre with water.

2.2.8 *Mixed indicator* — Weigh 0·08 g of naphthol green, 0·05 g orthocresol phthalein complexone, 0·06 g of eriochrome black T and 20 g ammonium chloride. Mix intimately by tritorating in an agate mortar.

2.2.9 *Standard EDTA solution* (0·025 *M*) — Dissolve 9 305 g of EDTA disodium salt in 1 litre of water, standardize EDTA solution against the standard calcium solution following the procedure for determination of calcium.

2.2.10 *Standard calcium solution* — Dissolve 5 g of calcium carbonate in 100 ml of water containing sufficient dilute hydrochloric acid (1 : 1) and boil to make a clear solution. Cool and dilute the solution to 100 ml in a volumetric flask, pipette out 25 ml in a 500 ml volumetric flask and dilute to the mark (1 ml = 0·001 4 g CaO).

2.2.11 *Sodium hydroxide solution* (5 *N*) — Dissolve 100 g of sodium hydroxide in water and dilute to 500 ml.

2.2.12 *P&R indicator* — Weigh 0·05 g of P & R (Patton and Reeder) indicator and mix intimately with 20 g of sodium chloride by triturating in agate mortar.

2.3 *Procedure*

2.3.1 Transfer two 50 ml aliquots of the main solution [*see* **2.3.3** of IS : 12107 (Part 2)-1987 Methods of chemical analysis of alumino silicate refractory materials: Part 2 Determination of silica] separately to two 250-ml conical flasks. Neutralize the solutions in both the flasks with

Adopted 24 August 1987

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sodium hydroxide solution in the presence of a small universal indicator paper. Acidify the two solutions to *p*H 3-4 by dropwise addition of dilute hydrochloric acid. Add 1 ml in excess. Add 10 ml (20 ml when alumina content is more than 40 percent) of triethanolamine solution to each flask and mix thoroughly. At this stage, the solution must be clear and alkaline indicated by the *p*H paper already present in. the solution. Add 5 ml of potassium cyanide solution. If any yellow colour develops due to iron, add 5 ml of hydroxylamine hydrochloride and boil till the solution is colourless. [If the titania content of the sample is high ($TiO_2 > 3$ percent), separate titania with 10 ml of hexamine solution].

2.3.2 Add 10 ml of buffer solution (*p*H 10) to a solution. Keep the volume of the solution to 100-150 ml add 0·2 g of mixed indicator and titrate immediately with standard EDTA solution till the colour of the solution changes from pink to bright green. Let the titre be '*a*' ml (for combined CaO + MgO).

2.3.3 Add to the other flask, 5 ml of sodium hydroxide solution and dilute to about 100-150 ml. Add 0·2 g of P & R indicator and titrate immediately with EDTA till the colour of the solution changes from pink to greenish blue. Let the titrate be '*b*' ml (for CaO only).

2.3.4 *Calculation*

$$\text{Calcium (as CaO), percent by mass} = \frac{b \times E_{\text{Ca}} \times 100}{M}$$

$$\text{Magnesium (as MgO), percent by mass} = \frac{(a - b) \times E_{\text{Mg}} \times 100}{M}$$

where

E_{Ca} = equivalent CaO, g/ml of EDTA;
E_{Mg} = equivalent MgO, g/ml of EDTA; and
M = mass in g of sample taken in the aliquot.

EXPLANATORY NOTE

Alumino silicate refractory materials contain alumina (Al_2O_3) and silica (SiO_2) in varying portions made synthetically by heating aluminium trifluoride at 1 000-1 200°C with silica and water vapour.

It is used in kilns and furnaces that operate at higher temperature or under conditions for which fire clay refractories are not suitable.

This standard has been prepared in different parts to cover the chemical analysis of various constituents in alumino silicate refractory materials. The other parts of the standard are:

Part 1 Determination of loss on ignition
Part 2 Determination of silica
Part 3 Determination of aluminium
Part 4 Determination of phosphorus
Part 5 Determination of titanium
Part 6 Determination of iron
Part 7 Determination of manganese
Part 9 Determination of sodium and potassium